Utilization of Lightweight Materials Made from Coal Gasification Slags

Quarterly Report September - November 1995

December 1995

Work Performed Under Contract No.: DE-FC21-94MC30056

For U.S. Department of Energy Office of Fossil Energy Morgantown Energy Technology Center Morgantown, West Virginia .

By Praxis Engineers, Inc. Milpitas, California



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1.0 PROJECT OBJECTIVES, SCOPE AND DESCRIPTION OF TASKS

1.1 <u>Introduction</u>

Integrated-gasification combined-cycle (IGCC) technology is an emerging technology that utilizes coal for power generation and production of chemical feedstocks. However, the process generates large amounts of solid waste, consisting of vitrified ash (slag) and some unconverted carbon. In previous projects, Praxis investigated the utilization of "as-generated" slags for a wide variety of applications in road construction, cement and concrete production, agricultural applications, and as a landfill material. From these studies, we found that it would be extremely difficult for "as-generated" slag to find large-scale acceptance in the marketplace even at no cost because the materials it could replace were abundantly available at very low cost. It was further determined that the unconverted carbon, or char, in the slag is detrimental to its utilization as sand or fine aggregate. It became apparent that a more promising approach would be to develop a variety of value-added products from slag that meet specific industry requirements. This approach was made feasible by the discovery that slag undergoes expansion and forms a lightweight material when subjected to controlled heating in a kiln at temperatures between 1400 and 1700°F. These results indicated the potential for using expanded slag as a substitute for conventional lightweight aggregates (LWA). The technology to produce lightweight and ultra-lightweight aggregates (ULWA) from slag was subsequently developed by Praxis with funding from the Electric Power Research Institute (EPRI), Illinois Clean Coal Institute (ICCI), and internal resources.

The major objectives of the subject project are to demonstrate the technical and economic viability of commercial production of LWA and ULWA from slag and to test the suitability of these aggregates for various applications. The project goals are to be accomplished in two phases: Phase I, comprising the production of LWA and ULWA from slag at the large pilot-scale, and Phase II, which involves commercial evaluation of these aggregates in a number of applications. Phase I was due to end on 14 December 1995 but has been extended to 14 June 1996 due to the unexpectedly long time it took for collection of slag samples. The scheduled completion date for Phase II has therefore been extended to 14 September 1997.

Primary funding for the project was provided by DOE's Morgantown Energy Technology center (METC) with significant cost sharing by Electric Power Research Institute (EPRI) and Illinois Clean Coal Institute (ICCI).

1.2 Scope of Work

The project scope consists of collecting a 20-ton sample of slag (primary slag), processing it for char removal, and subjecting it to pyroprocessing to produce expanded slag aggregates of various size gradations and unit weights, ranging from 12 to 50 lb/ft³. A second smaller slag sample will be used for confirmatory testing. The expanded slag aggregates will then be tested for their suitability in manufacturing precast concrete products (e.g., masonry blocks and roof tiles) and insulating concrete, first at the laboratory scale and subsequently in commercial manufacturing plants. These products will be evaluated using ASTM and industry test methods. Technical data generated during production and testing of the products will be used to assess the overall technical viability of expanded slag production.

In addition, a market assessment will be made based on an evaluation of both the expanded slag aggregates and the final products, and market prices for these products will be established in order to assess the economic viability of these utilization technologies. Relevant cost data for physical and pyroprocessing of slag to produce expanded slag aggregates will be gathered for comparison with (i) the management and disposal costs for slag or similar wastes and (ii) production costs for conventional materials which the slag aggregates would replace. This will form the basis for an overall economic evaluation of expanded slag utilization technologies.

1.3 <u>Task Description</u>

A summary of the tasks to be performed in Phase I is given below:

- Task 1.1 Laboratory and Economic Analysis Plan Development: Development of a detailed work plan for Phase I and an outline of the Phase II work.
- Task 1.2 Production of Lightweight Aggregates from Slag: Selection and procurement of project slag samples, slag preparation including screening and char removal, and slag expansion in direct- and indirect-fired furnaces. Preliminary laboratory-scale studies will be conducted before bulk samples of expanded slag are collected for processing. The char recovered from the slag preparation operation will be evaluated for use as a kiln fuel and gasifier feed. Environmental data will also be collected during preparation and expansion of slag.
- Task 1.3 Data Analysis of Slag Preparation and Expansion: Analysis and interpretation of project data, including development of material and energy balances for slag processing and product evaluation.
- Task 1.4 Economic Analysis of Expanded Slag Production: Economic analysis of the utilization of expanded slag by determining production costs for slag-based LWAs and ULWAs. An estimated market value will be established for the various expanded slag products. Expanded slag production costs will be compared with the costs of disposal and management of slag as a solid waste.
- **Task 1.5 Topical and Other Reports**: Preparation and delivery of topical, financial status, and technical progress reports in accordance with the Statement of Work.

The Phase II effort will focus on field studies conducted on expanded slag aggregates to test their performance as substitutes for conventional materials in various applications, including masonry blocks, roof tiles, insulating concrete, and insulation fill. Mix designs will be formulated and tested by refining the material proportions used in previous work. Commercial manufacturing practices, standards, and equipment will be used for this work. New applications may also be identified during the course of this work. The economic analyses conducted in Phase I will be further refined in Phase II using the new data.

1.4 Scope of this Document

This is the fifth quarterly report and summarizes the work undertaken during the performance period between 1 September 1995 and 30 November 1995.

2.0 SUMMARY OF WORK DONE DURING THIS REPORTING PERIOD

2.1 Summary of Major Accomplishments

The following was accomplished during the current reporting period:

- 1. The primary slag sample was screened into three size fractions. The 1/4" x 10M and 10 x 50M fractions were expanded in discrete particle form, and the minus 50M slag was pelletized along with an expansible clay binder before pyroprocessing.
- 2. The minus 50M slag was pelletized using slag:clay ratios of 80:20 and 50:50. A batch of clay pellets was also made to serve as a control sample.
- 3. Fuller commissioned two direct-fired kilns (3-ft dia x 30-ft long and 1-ft dia x 15-ft long) including gas analysis, and pollution control equipment. The smaller kiln was selected because its lower feed rate permitted study of the expansion characteristics of Slag I pelletized fines and Slag II, which were available in small quantities.
- 4. Early on 11/13/95 the kiln was preheated and the feed was started. The test runs consisted of:
 - Temperature vs. density studies
 - Production runs to produce LWA products at 30, 40 and 50 lb/ft³ using 1/4" x 10M, 10 x 50M and 1/4" x 50M slag. Also, extruded pellets containing 20%, 50% and 100% clay were expanded to the same three unit weights.
- 5. The 3-ft diameter kiln was operated for three days for nearly 16 hours per day to complete the slag expansion production runs for Slag I, the primary slag.
- 6. The 1-ft. diameter kiln was operated on November 15-16 to process the Slag I pelletized fines and Slag II, to accommodate temperature studies at a lower feed rate.
- 7. Expanded slag products were sampled every 15 minutes to correlate the temperature vs. density variability. The products were stored in drums as a function of real time.
- 8. Fuller's fluid bed calciner was also commissioned for use. This equipment is considered more suitable for producing ultra-lightweight aggregates (ULWA) because the feed is not directly exposed to the flame.

The kiln testing program was completed successfully and the products are being characterized as per ASTM standards. Testing with the fluid bed calciner will continue in order to make a product with a unit weight equivalent to the classification for ULWA (less than 12 lb/ft³).

2.2 Chronological Listing of Significant Events in This Quarter

The following significant events occurred during the current reporting period:

Date	Description		
9/10/95	Laboratory studies of slag expansion completed		
9/20/95	Laboratory testing of pelletized slag/clay started		
10/15/95	1-ft and 3-ft diameter direct-fired kilns commissioned for pilot testing		
11/15/95	Pilot testing of Slag I in the 3-ft dia. direct-fired kiln completed		
11/16/95	Pilot testing of pelletized Slag I in the 1-ft dia. direct-fired kiln completed		
11/17/95	Pilot testing of Slag II in the 1-ft dia. direct-fired kiln completed		

3.0 TO DATE ACCOMPLISHMENTS

This section summarizes the work completed to date in the first five quarters of the project:

Date	Accomplishments .			
10/24/1994	"Draft Laboratory and Economic Analysis Plan" prepared and submitted			
11/07/1994	Advance sample of primary project slag collected for testing for char removal			
11/18/94	Slag processing for char removal completed successfully on the advance sample and prepared slag sent to Fuller and Silbrico for expansion testing			
12/02/94	Final "Laboratory and Economic Analysis Plan" prepared and submitted			
12/14/94	Testing of advance slag sample at Fuller and Silbrico indicates that it has excellent expansion properties			
02/15/94	Evaluation of blendability of slag fines with an expansible clay initiated at Fuller			
03/05/95	Decision made regarding primary and secondary project slag samples			
04/15/95	Laboratory extrusion testing of the advance sample using an expansive clay binder completed at Fuller			
05/21/95	Primary slag sample received at Penn State for preparation			
05/30/95	Characterization of primary slag using samples from two drums completed			
06/01/95	Pilot unit for char removal set up			
06/21/95	Second slag sample tested and evaluated for expansion characteristics			
06/30/95	Char removal operational problems solved and continuous slag processing started			
08/20/95	Primary slag sample processing for char removal completed			
08/31/95	Slag screening of primary slag at 10 mesh and 50 mesh started			
9/10/95	Laboratory studies of slag expansion on the two slags completed			
9/20/95	Laboratory testing of pelletized slag/clay blends started			
10/15/95	1-ft and 3-ft diameter kilns commissioned for pilot testing			
11/15/95 Pilot testing of Slag I in 3-ft dia. direct-fired kiln completed				
11/16/95	Pilot testing of pelletized Slag I in 1-ft dia. direct-fired kiln completed			
11/17/95	Pilot testing of Slag II in 1-ft dia. direct-fired kiln completed			

4.0 TECHNICAL PROGRESS REPORT

The major accomplishment of this reporting period was expansion testing of slags in the direct-fired kilns. The data available from the laboratory expansion tests conducted earlier using both the primary slag (Slag I) and the second slag (Slag II) in discrete particle and pelletized form constituted the basis of this work. Based on these results, pilot testing for production of LWA from both slag samples was planned and implemented in this reporting period. Prior to pilot testing, the two direct-fired pilot kilns and ancillary equipment were commissioned. The slag processed for char recovery was also screened into 1/4" x 10M, 10 x 50M, and minus 50M size fractions. The two coarse fractions were used directly as kiln feed. The minus 50M slag fines

were extruded to make pellets using an expansive clay binder in proportions determined during laboratory-scale tests. Using the two direct-fired kilns, large quantities of LWA products with unit weights in the 30-50 lb/ft 3 range were made from the 1/4" x 10M and 10 x 50M size fractions and the pelletized slag fines from Slag I. LWA products were also made from Slag II. Laboratory evaluation and characterization of these products is in progress. The bulk of the products will be used for commercial-scale evaluation in various applications in Phase II. Details of the work done in this reporting period are given in the following sections.

4.1 Objectives of Pilot Test Program

The objectives of the pilot slag expansion test program, based on the Phase I Work Plan, were to:

- 1. Demonstrate the production of LWAs from slag and pelletized slag in a direct-fired pilot-scale 3-ft x 30-ft rotary kiln. This consisted of the following steps:
 - Demonstrate production of LWAs from slag with unit weights in the 30-50 lb/ft³ range by controlling the hot zone temperature. Study the temperature vs. product unit weight relationship.
 - Produce a large batch of LWAs for laboratory and commercial evaluation from the +10M and the 10 x 50M slag fractions and the pelletized minus 50M slag, with product unit weights of 30, 40, and 50 lb/ft³
 - Explore the potential for reducing product unit weights below 30 lb/ft³.
- 2. As part of developing the process engineering design, collect the following technical data:
 - Determine the feed rate, residence time, energy consumption, and other design data for commercial sizing of the direct-fired kiln for processing of slag
 - Determine a mass balance for direct-fired kiln operation
 - Sample the off-gas stream to determine SO₂, NO_x, CO, CO₂, and O₂
 - Conduct laboratory evaluation of the kiln products to confirm their suitability for various applications. Conduct physical, chemical, and environmental characterization of the expanded slag products.

4.2 Pilot Test Runs for Direct-Fired Kilns

In order to achieve the project objective of demonstrating the production of expanded slag at the pilot scale, a test matrix was developed for the direct-fired kiln to generate products with unit weights of 30-50 lb/ft³, which correspond to the category of LWAs.

Table 1 lists the slag feed materials to be processed in the two direct-fired kilns and the indirect kiln/fluid bed calciner as well as the target products.

Table 1. Slag Quantities to be Processed During Pilot Tests

Slag/Size	Direct-Fired Kilns for LWA Production		Indirect-Fired Kiln or Fluid Bed for	Total, drums
	3-ft dia.	1- dia.	ULWA Production ³	
Slag I: 1/4" x 10M	10 drums 30, 40, 50 lb /ft³		2 drums 12 lb/ft ³	12
Slag I: 10 x 50M	9 drums 30, 40, 50 lb/ft ³	-	2 drums 12 lb/ft³	11
Slag I: 1/4" x 50M	1 drum 30, 40, 50 lb/ft ³			1
Slag I: -50M pellets slag 80/20 clay 50/50 0/100		30, 40, 50 lb/ft ³ 1 drum 1 drum ½ drum	50 lb/ft³ ½ drum 	3
Slag II: +10M1		1½ drums		1½
Slag II: -10 M			2 drums 30, 40, 50 lb/ft ³	2
Slag III: -10M²			1 drum 40 lb/ft ³	1
Total drums @ 600 lb each	20	4	6½	31½
Feed rate, lb/hour	500-750	50	40	
Operating time, hours	24	72		

Explanations:

- (1) Slag II (TVA slag): The +10M slag was expanded. The -10M slag will need to be screened at 50M to remove the fines and then expanded in a fluid bed calciner.
- Slag III (Sufco slag): Earlier tests with this slag indicated potential fusion problems. It may need to be tested further in the laboratory before pilot testing in the fluid bed calciner.
- (3) A fluid bed calciner may be used in place of an indirect-fired furnace if it provides good process control. This work is planned for the next quarter.

4.3 Pilot Operation of Large Direct-Fired Kiln

A 3-ft x 30-ft direct-fired rotary kiln was commissioned prior to conducting pilot expansion tests for the primary slag (Slag I). The kiln rotational speed was set at 2.6 rpm to provide a total residence time of 30 minutes. This was based on a kiln slope of 3/8"/ft and an angle of repose

of 35° for the material. Fuel Oil No. 2 was selected as the kiln fuel for this program. The kiln was started at midnight on 11 November 1995 and the fuel oil burner was adjusted to obtain a starting hot zone temperature of 1500°F (816°C), as indicated by the hot zone temperature indicator. The kiln was preheated for 6 hours. Based on the fuel oil flow rate, a kiln exit oxygen concentration of 10%, and exit gas temperature, the kiln exit gas velocity was calculated at 15 ft/sec and the draft was adjusted accordingly.

The belt feed system was set to deliver a slag feed rate of 500 lb/hour using the $10 \times 50M$ fraction of Slag I. This feed rate provides a sufficiently long operating time for Slag I to generate all required operating data including the temperature vs. density relationship. The peak feed rate for this kiln is up to 1000 lb/hr.

Upon achieving the desired temperature conditions, the kiln feed was started at 8 a.m. on 13 November 1995, using the 1/4" x 10M size fraction of the primary slag (Slag I). After allowing the operating conditions to stabilize for 30-60 minutes, product samples were taken every 15 minutes to measure the unit weight. The kiln hot zone temperature was adjusted as necessary to obtain a product unit weight of 50 lb/ft³. Once the proper unit weight was obtained, steady-state conditions were maintained for a period of 30-60 minutes before any changes were made in the temperature or feed rate. Temperature changes were generally limited to 15-20°F increments and maintained for 60 minutes. This procedure was followed, increasing the temperature until a product unit weight of 30 lb/ft³ was achieved.

Upon concluding operations at conditions necessary to produce a product with a unit weight of 30 lb/ft³, the temperature was decreased to obtain conditions required to produce a 40 lb/ft³ product using the 10 x 50M fraction of Slag I. Steady-state conditions were maintained until all of the slag was processed, while checking the product unit weight every 30 minutes as part of operational quality control. All operating conditions were recorded every 30 minutes. Tests were also successfully run using a mix of these two slag fractions, i.e., a 1/4" x 50M feed. This approach simplifies the slag screening procedure prior to expansion.

Operating information was collected and recorded for each production run or phase, defined as expansion of a slag size fraction (e.g., Slag I, $10 \times 50M$) to produce a specific unit weight product (say, 30 lb/ft^3). The expanded slag products were collected and stored in separate drums for each phase labeled with information including the operating hours. The kiln shell temperature was recorded 1-2 times per operating phase. Samples of the off-gas stream were analyzed to determine SO_2 , NO_x , CO, CO_2 , and O_2 at the stack.

The kiln was operated for nearly 16 hours/day for the first day and 10 hours/day for the next two days to complete the slag expansion production runs for Slag I at 30, 40 and 50 lb/ft³ unit weights. Product samples collected for quality control were used to determine the unit weight and then stored in separate bags and labeled for further analysis. The bulk of the product material was stored in product drums which were weighed and labeled. The label information included sample identification, test batch, product unit weight, and the production time. This information was also recorded on the product sheets. The kiln dropout was collected, weighed, and recorded every 30 minutes. The baghouse material was sampled and weighed every 60 minutes, then tagged and recorded. The dropout material was also sampled every 60 minutes for further analysis. In addition, a feed sample was collected for each production run.

4.4 Pilot Operation of Small Direct-Fired Kiln

The 1-ft x 15-ft direct-fired rotary kiln was started on 15 November 1995 to conduct pilot expansion tests to process the second slag (Slag II) and extruded pellets made from the minus 50M fines from Slag I. The smaller kiln was selected because it can be operated at a lower feed rate (50-100 lb/hour) thus increasing the operating period. This, in turn, allows expansion studies to be conducted while requiring smaller quantities of feed materials thus conserving the project slag samples.

The kiln rotational speed was set at 2.4 rpm to provide a total residence time of 30 minutes. This was based on a kiln slope of 1/2"/ft and an angle of repose of 40° for the pelletized material. Fuel Oil No. 2 was selected as the kiln fuel for this program. The kiln was started early on 16 November 1995. The fuel oil burner was adjusted to obtain a starting hot zone temperature of 1900°F (1038°C) as indicated by the hot zone temperature indicator. The draft was adjusted to obtain a kiln exit gas oxygen concentration of approximately 10%. The kiln was preheated for approximately 3 hours. Based on fuel oil flow rate, a kiln exit oxygen concentration of 10%, and the exit gas temperature, the kiln exit gas velocity was calculated at 15 ft/sec and the draft was adjusted accordingly.

The vibratory feed system was set to deliver a rate of 50 lb/hour using 80/20 slag/clay pellets made from the Slag I minus 50M fines. This feed rate provides a sufficiently long operating period to generate all required operating data including the temperature vs. density relationship.

The operating information was collected and recorded for each production run or phase, defined as expansion of a slag size fraction (e.g., Slag I, 80/20 pellets) to produce a specific unit weight product (say, 30 lb/ft³). The expanded slag products were collected and stored in separate drums for each phase, labeled with of the operating hours, for future reference. The kiln shell temperature was recorded 1-2 time per operating phase. Samples of the off-gas stream were analyzed to determine SO_2 , NO_x , CO, CO_2 , and O_2 at the stack using the same equipment as was used for the larger kiln.

Procedures for kiln operation, product quality monitoring, and data gathering were identical to those used for the larger kiln.

4.5 Preliminary Tests Using a Fluid Bed Calciner to Produce ULWA

In order to produce ultra-lightweight aggregates (ULWA) from slag, Praxis conducted expansion tests with the TVA slag in an indirect-fired furnace under previous projects funded by EPRI and ICCI, respectively. Earlier in the current project, a laboratory-scale indirect-fired rotary kiln was used to test the feasibility of expanding Slag I. The results, reported in Quarterly Report No. 2, indicated that products with unit weights as low as 22 lb/ft³ could be obtained. Lower unit weights can be achieved with indirect expansion because the flame does not come into direct contact with the material, thus affording better temperature control even at or near the fusion point of the slag. However, since this method involves heating the material indirectly by heating the kiln shell, energy consumption tends to be very high. This may make the use of an indirect-fired kiln uneconomical for commercial production of expanded slag aggregates.

Fuller Company has recently developed a fluid bed calciner in which the flame does not come into direct contact with the slag. This method provides the potential for producing ULWA in an energy efficient manner and may also be used to produce LWA. Praxis conducted preliminary tests using a fluid bed calciner and the initial results were very promising. Based on these results, we are considering testing the production of ULWA in the fluid bed calciner rather that an indirect-fired rotary kiln as stated in the project Work Plan.

4.6 Conclusions and Recommendations

The major accomplishments in the current reporting period were pilot-scale testing and demonstration of the production of LWAs from slag and of extruded pellets made from slag fines. Two size fractions (1/4" x 10M and 10 x 50M) of the primary slag sample were processed directly. The minus 50M fraction was expanded following pelletization with an expansive clay binder using an extruder. Two ratios of slag:clay were used: 80:20 and 50:50. An all-clay pellet batch was also expanded to produce a conventional aggregate to serve as a control sample. The second slag was also tested in discrete particle form at +10M.

The following conclusions were drawn from the work done during this reporting period:

- The 1/4" x 10M and 10 x 50M slag fractions were expanded in discrete particle form in a pilot-scale direct-fired rotary kiln using Slag I to produce LWA at unit weights ranging between 30 and 50 lb/ft³. These results confirmed the laboratory-scale results.
- Temperature vs. density studies were conducted and product unit weights could be varied in the 30-50 lb/ft³ range by means of temperature control. However, further reduction of the unit weight below 30 lb/ft³ was not feasible due to potential fusion problems.
- Similar results were achieved for extruded slag pellets made from minus 50M slag fines.
- The feed particle size to the 1-ft and 3-ft diameter direct-fired kilns could be changed gradually without causing problems and the product unit weight specifications could also be changed without causing operating problems.
- The 3-ft dia. kiln was also tested successfully using a mixed feed consisting of a blend of the 1/4" x 10M and 10 x 50M fractions. This run demonstrated that the slag feed does not need to be screened at 10M for expansion.
- Fuller's fluid bed calciner was commissioned for testing with the objective of producing lower-unit-weight aggregates than those produced from the direct-fired kiln. The initial results were very successful. This approach may provide a more suitable method for producing ultra-lightweight aggregates than the indirect-fired kiln.

The pilot-scale direct-fired kiln testing program was completed successfully. The products are being characterized as per ASTM standards.

The following recommendations are made based on the work completed in this reporting period:

The feasibility of producing expanded slag aggregates with unit weights below the classification for conventional LWAs (i.e., ULWAs) should be explored using a fluid bed calciner rather than an indirect-fired rotary kiln.

5.0 PLAN FOR THE NEXT QUARTER

The following activities are planned for the next quarter:

- Laboratory evaluation of expanded products from the pilot-scale tests
- Data analysis of the kiln process for producing LWAs using discrete-particle slag fractions and extruded pellets
- Analysis of operational data to establish mass and material balances for slag processing
- Evaluating the production of ULWA
- Work on economic analysis will continue.

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